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COMPARISON OF TWO INSTRUMENTS FOR DETERMINING HARDNESS OF ELASTOMERS

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ABSTRACT

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To reach a higher degree of accuracy in control evaluations of rubber compounds, a comparison was made of two commercially available instruments for measuring hardness of elastomeric compounds. These instruments, the Shore durometer and ASTM (Tinius Olsen), were compared over a wide hardness range on 13 types of rubber formulations.

Studies indicated that,although the ASTM (Tinius Olsen) instrument requires a more refined test specimen and is somewhat more difficult to operate, it is a more precise instrument and should be used where very close tolerances are involved or as a "referee" in case of doubt with other instruments. The Shore durometer provides a rapid means for measuring hardness of elastomers; the specimen size is not critical; and the Shore durometer accuracy is sufficient for control evaluations as well as for the majority of end items.

With the graphs and tables in this report, it is possible to convert units of measure from one instrument to the other for a particular compound of interest; however, to prepare a single table (or graph) illustrating a "typical" correlation for all elastomers is not practical because of the variation in creep with different formulations.

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MATERIALS DIVISION
PROPULSION AND VEHICLE ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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SUMMARY

To reach a higher degree of accuracy in control evaluations of rubber compounds, a comparison was made of two commercially available instruments for measuring hardness of elastomeric compounds. These instruments, the Shore durometer and ASTM (Tinius Olsen), were compared over a wide hardness range on 13 types of rubber formulations.

Studies indicated that, although the ASTM (Tinius Olsen) instrument requires a more refined test specimen and is somewhat more difficult to operate, it is a more precise instrument and should be used where very close tolerances are involved or as a "referee" in case of doubt with other instruments. The Shore durometer provides a rapid means for measuring hardness of elastomers; the specimen size is not critical; and the Shore durometer accuracy is sufficient for control evaluations as well as for the majority of end items.

With the graphs and tables in this report, it is possible to convert units of measure from one instrument to the other for a particular compound of interest; however, to prepare a single table (or graph) illustrating a "typical" correlation for all elastomers is not practical because of the variation in creep with different formulations.

INTRODUCTION

The purpose of this investigation was to compare the relative merits of the ASTM (Tinius Olsen) hardness instrument with the Shore "A" durometer for use in research work involving rubber compositions and to render a correlation between them. Because it has been reported in previously published papers, this investigation did not consider the theory of hardness measurements by the type of instruments under discussion.

The determination of hardness by any process or instrument is without absolute units of measure (the results are empirical). Hardness is more commonly defined as a quality or state of being hard and may be determined by one of the following methods: (1) the relative ability of one material to scratch the surface of another; (2) the relative resistance to surface indention. Although the first method is not applicable to rubber, the second is well suited to the measurement of hardness in rubber and rubber-like materials, including some plastics.

There are numerous types of surface indention instruments now being used in the commercial rubber industry. The accuracy of readings or measurements obtained with these instruments is directly related to the design and calibration of the instrument and to the skill of the operator. Two of the more acceptable types of indention instruments were compared over an extensive range of hardness in order to select the one that offered a higher degree of accuracy and reproducibility. Approximately 1600 measurements were made during this study. These instruments and a description of their merits are given below.

Shore Durometer

Three versions of the durometer ("A" Scale) which have a similar basic operation are commercially available. These instruments are spring loaded and depend upon the depth of penetration of a truncated cone as an indication of hardness. The design details and methods describing the use of these instruments are listed in ASTM Method D676-59T.

The least accurate of the durometers is the Rex Gage (FIG 1A). This instrument is graduated in increments of five points, which limits

its usage. An improvement over the Rex Gage is the Shore A Durometer (FIG 1B). This instrument is also graduated in five-degree increments; however, the increments are separated over a 90° circle dial, and fairly accurate point readings may be taken. A much improved Shore A instrument is the A-2 Model (FIG 1C) which is graduated in one-degree increments over a 270° circle dial. This instrument was selected as the more accurate of the Shore durometers and was used as one of the instruments for comparison in this report.

ASTM (Tinius Olsen) Instrument

The ASTM instrument (FIG 1D) operates under a dead-weight load principle, having a total angular deflection of 790° and is described in ASTM method D314-58. This instrument is capable of producing a more precise reading than the Shore durometer; however, its numerical scale is quite unlike that of the Shore durometer, and its usage of specimen size is limited. Since most rubber technologists express hardness in "Shore A" units, it was believed that a correlation curve could be established for particular compounds to convert hardness readings from the ASTM scale to the Shore A scale where precise control measurements were necessary.

EXPERIMENTAL

All tests were made according to the ASTM specifications for each instrument, except for variations in "time lapse" on the Shore durometer, which will be explained in the next paragraph. All specimens were formulated in the Non-Metallic Materials Branch and were vulcanized in accordance with standard formulation procedures. Due to the variation of creep in many elastomers, it was decided that each of the compounds should be studied at various hardness levels to compare accurately the two instruments.

An average of 10 readings with each instrument was taken for each of the compounds listed in Table I. The results of these tests are shown graphically in FIG 2, where a complete comparison of hardness is given for the 13 types of elastomers that were used in the study. As mentioned above, it was necessary to vary the reading time on the Shore durometer because of the creep observed when initial contact to the specimen was made with the indention point of the instrument. The ASTM procedure (D676-59T) does not specify the exact time lapse before reading the Shore durometer. With some operators this lapse may be 15 to 30 seconds, or a reading may be made upon initial contact to the specimen. To show the amount of creep for each of the compounds measured by the A-2 durometer, an initial contact reading (zero second) was made as well as a 30-second time lapse. (This was not necessary for the ASTM instrument since the ASTM procedure specifies a 30-second time lapse before reading) These data are shown graphically in FIG 3 through 15 and illustrate the amount of creep recorded between 0 and 30 seconds for the various elastomers. It was observed throughout the study that although some of the compounds followed a characteristic pattern (when plotted on linear paper) there was a noticeable variation from this pattern with nitrile, Viton, Urethane, Thiokol, and Kel-F elastomers. These elastomers showed a pronounced drift during a 0- to 15-second time interval and a significant amount of creep from 15 to 30 seconds (FIG 3 through 15). Generally, it was observed that the overall average creep was more significant at the higher hardness levels than it was at the lower hardness stocks (Table II).

FIG 16 shows that the average results for the 13 types of rubber tested give a differential of approximately five Shore A hardness units when readings are taken at 0- and 30-second intervals. An intermediate reading at 15 seconds is also indicated on this graph and illustrates the manner in which the rate of creep is diminishing.

A careful analysis of the data obtained after a 30-second time lapse on the Shore A-2 instrument coincided closely with the values obtained on the same elastomers with the ASTM instrument. A curve was prepared (FIG 17) from which readings that were taken with the ASTM instrument may be converted to Shore A units for a 30-second reading.

It was observed that although some elastomers may have the same Shore A hardness there is a slight variance in the ASTM hardness of these same compounds. This is believed to be caused by the higher pressure which is applied to the specimen by the ASTM instrument, resulting in a slightly larger amount of drift during the final 15 seconds of the test. Again, the inherent properties of the polymer tend to control the amount of evident variation.

To determine the effect of the state-of-cure on the drift characteristic of the elastomer, three compounds were selected to be cured to different levels and tested at various hardness levels. For this study, one compound having the highest drift rate (nitrile rubber) and two having a low drift rate (neoprene and natural) were chosen. The cure levels and the results of the tests which were made were as follows:

Nitrile. - Creep was greatest for nitrile type rubber, and this effect is shown graphically in FIG 18, where readings were taken at 0-, 15-, 30-, and 60-second intervals with the Shore A-2 and at 30 seconds with the ASTM instrument.

This elastomer was tested at all hardness levels ranging from Shore 40 through 90 at cure times of 30, 60, and 90 minutes at 143°C (289°F). The level of cure had essentially no effect on the hardness of this elastomer, showing a variation of ± 1 point at all ranges when compared to the hardness at the standard cure of 60 minutes at 143°C (289°F). These results are shown in Table III.

Neoprene. - This elastomer was tested at all hardness levels ranging from Shore 30 through 90, each at cure times of 15, 30, and 45 minutes at 153°C (307°F). As with nitrile, there was no adverse effect on the hardness of this elastomer due to the time of cure. Variations observed at all levels tested were within ± 2 points of hardness values obtained at the standard cure time of 30 minutes at 153°C (307°F). These results are shown in Table IV.

Natural. - This elastomer was tested at all hardness levels ranging from Shore 30 through 80; each was cured at times of 10, 20, and 30 minutes at 143°C (289°F). There was a distinct drop (up to 5 points) in hardness at the 30-minute cure rate, particularly in the lower and medium hardness regions (Shore 30 through 60). The 10-minute cure at 143°C (289°F) gave higher hardness than the 20- to 30-minute cures; it is believed that reversion of the polymer was responsible for this observed effect. This variation, however, is not considered to be significant since the amount of change was ± 2 points throughout the entire hardness range when compared to tests at the standard cure rate of 20 minutes at 143°C (289°F). The higher hardness compounds, Shore 70 and 80, showed essentially no change in hardness by varying the length of cure.

From the data, it may be concluded that the characteristic or formulation of the elastomers is probably more responsible for the amount of creep observed than the level of cure.

CONCLUSIONS

The Shore A-2 durometer provides a rapid means of measuring hardness in elastomers, and its accuracy is sufficient in the majority of end items and routine control work. Variations between operators and laboratories possibly account for a substantial amount of error due to pressure applied to the instrument, time of lapse during readings, and whether it is hand held or mounted on a stand. However, this amount of variation can be eliminated essentially by using a "standard practice routine."

The ASTM (Tinius Olsen) instrument is somewhat more difficult to operate and requires a more refined specimen for test; however, it is a more precise instrument and should be used if very close tolerances must be observed or as a referee in case of doubt with the Shore durometer.

Because of the variation in creep with different formulations, the direct comparison of instruments would be difficult unless the same formulation was used for each test. With the graphs and tables included in this report (FIG 3 through 15), it is possible to obtain an accurate correlation for a particular compound of interest. FIG 17 illustrates an approximate correlation of "Shore vs ASTM Hardness" for 13 formulations and may be used where only a relative amount of accuracy is required.

TABLE 1

ASTM VS SHORE HARDNESS FOR THIRTEEN RUBBER COMPOUNDS

NOMINAL HARDNESS*	30		40		50		60		70		80		90	
HARDNESS BY METHOD	A**	B***	A	B	A	B	A	B	A	B	A	B	A	B
COMPOUND														
Natural	175	29	144	34	103	43	68	54	41	67	21	80		
Neoprene	148	32	107	39	77	49	56	59	39	70	27	78	20	87
Hypalon							65	56	47	66	35	75		
BN			201	25	146	34	100	43	64	52	47	60	29	73
Silicone (RA 28)	131	26	126	34	84	45	70	52	51	62	28	77		
Kel-F							62	56	42	66	33	72		
BS	166	28	112	38	82	47	59	56	39	68	27	76	22	81
LS							60	57	48	64	35	74		
Acrylic			103	40	81	48	65	54	51	61	29	69		
Thiokol							70	53	46	63	30	75		
Butyl					87	48	64	59	47	68				
Urethane							66	53	39	67				
Viton-A							74	55	46	68	34	75	22	83

* Nominal Shore Hardness
** Tinius Olsen Hardness Instrument (ASTM D314-58)
*** Shore A-2 Durometer (ASTM D676-59T) (at 30 seconds)

TABLE II

AVERAGE* SHORE AND ASTM HARDNESS OF THIRTEEN STANDARD COMPOUNDS AT ALL HARDNESS LEVELS

Nominal Hardness (Shore)	Actual Shore A-2 @			Actual ASTM @ 30 Sec
	0 Sec	15 Sec	30 Sec	
30	29	29	29	155
40	38	36	35	132
50	48	46	45	94
60	58	55	54	68
70	69	65	65	46
80	78	75	74	31
90	86	82	81	23

*Average data were taken from 10 readings on each of the thirteen standard compounds at each hardness level available.

TABLE III

HARDNESS VALUES OF NBR RUBBER AT CURE LEVELS OF 30', 60', AND 90' AT 143°C (289°F)

(Shore)

Nominal Hardness (Shore)	Actual Shore A-2 Hardness (30 Sec)		
	30'	60'	90'
40	20	21	21
50	33	34	32
60	41	41	43
70	51	52	52
80	62	63	62
90	75	73	74

(ASTM)

Nominal Hardness (Shore)	ASTM Hardness		
	30'	60'	90'
40	215+	215+	215+
50	161	158	155
60	105	105	103
70	71	70	70
80	44	44	44
90	25	25	25

TABLE IV

HARDNESS VALUES OF NEOPRENE RUBBER AT CURE LEVELS OF 15', 30', AND 45' AT 153°C (307°F)

(Shore)

Nominal Hardness (Shore)	Actual Shore A-2 Hardness (30 Sec)		
	15'	30'	45'
30	31	33	33
40	40	40	40
50	50	51	51
60	62	61	61
70	70	70	70
80	78	79	79
90	88	87	90

(ASTM)

Nominal Hardness (Shore)	ASTM Hardness		
	15'	30'	45'
30	154	157	157
40	120	119	117
50	78	78	78
60	54	54	55
70	40	39	39
80	30	29	27
90	17	18	17

TABLE V

HARDNESS VALUES OF NATURAL RUBBER AT CURE LEVELS OF 10', 20', AND 30' AT 143°C (289°F)

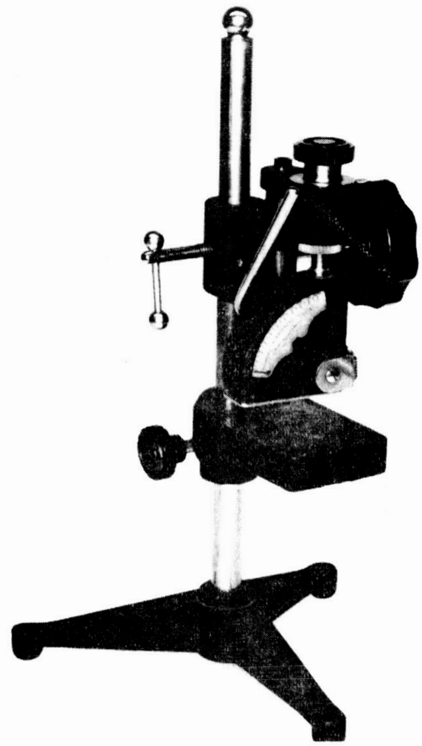
(Shore)

Nominal Hardness (Shore)	Actual Shore A-2 Hardness (30 Sec)		
	10'	20'	30'
30	31	29	26
40	37	36	34
50	49	49	47
60	57	56	52
70	80	82	81
80	81	82	81

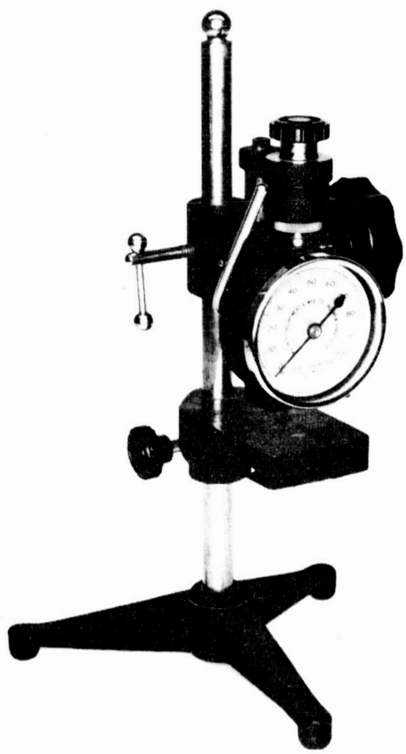
(ASTM)

Nominal Hardness (Shore)	ASTM Hardness		
	10'	20'	30'
30	171	187	206
40	141	141	161
50	88	90	95
60	64	73	82
70	24	22	23
80	21	25	30

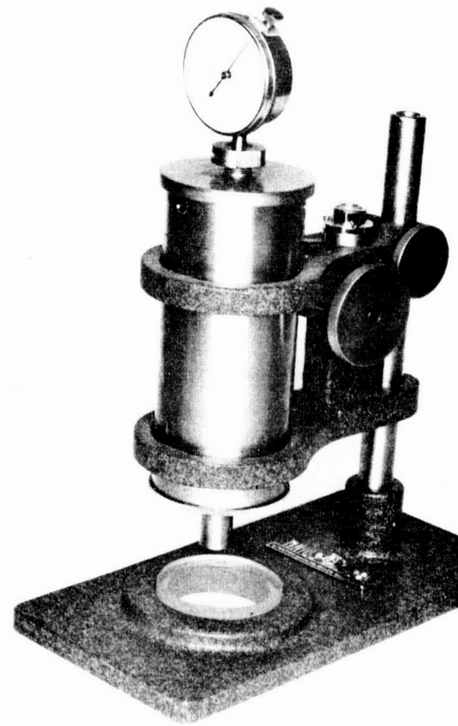
A. REX GAGE - TYPE A



B. SHORE DUROMETER - TYPE A



C. SHORE DUROMETER - TYPE A-2



D. TINIUS OLSEN TESTER - ASTM

FIGURE 1. HARDNESS MEASURING INSTRUMENTS

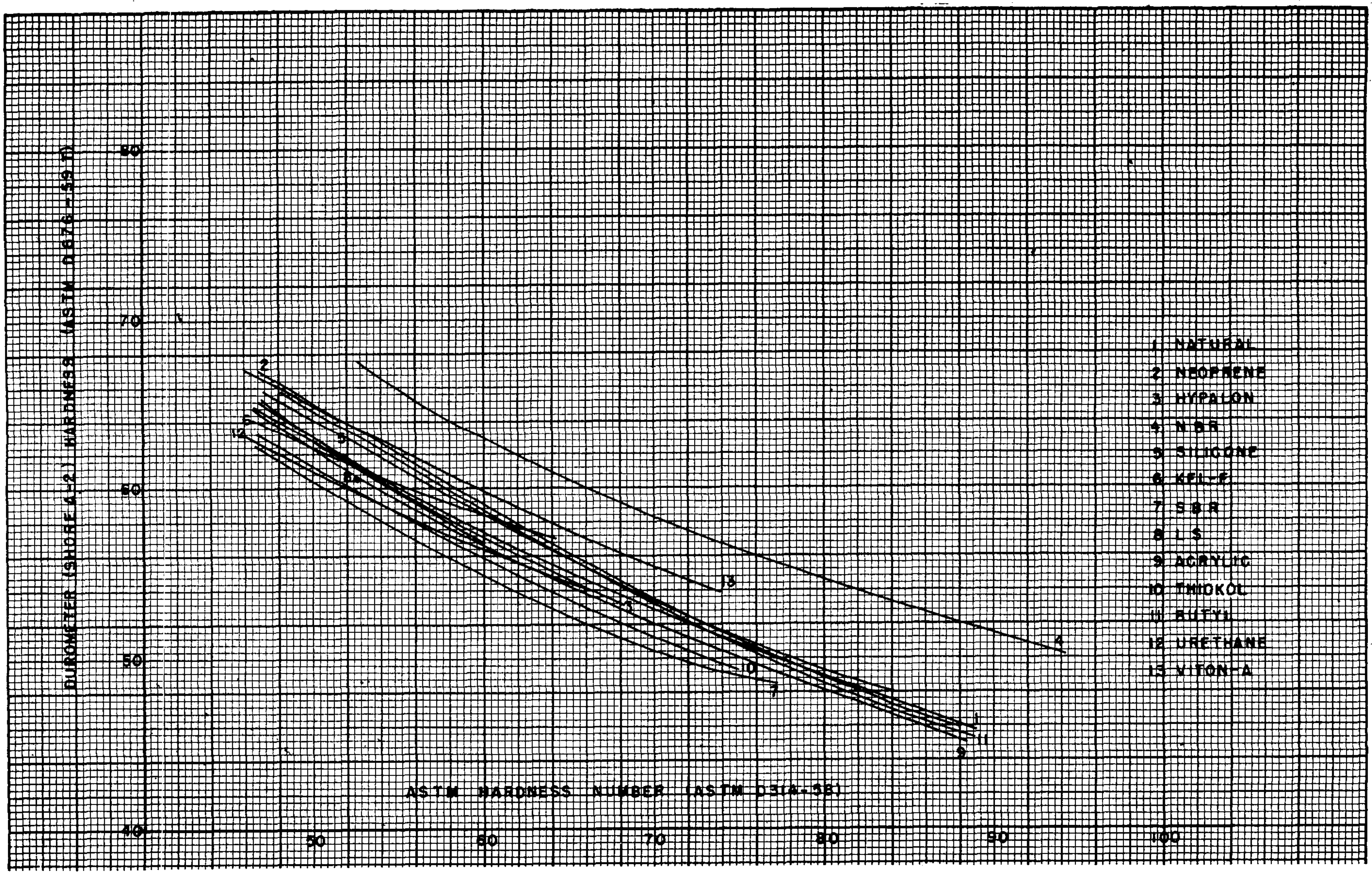


FIGURE 2 ASTM VS SHORE HARDNESS OF THIRTEEN COMPOUNDS

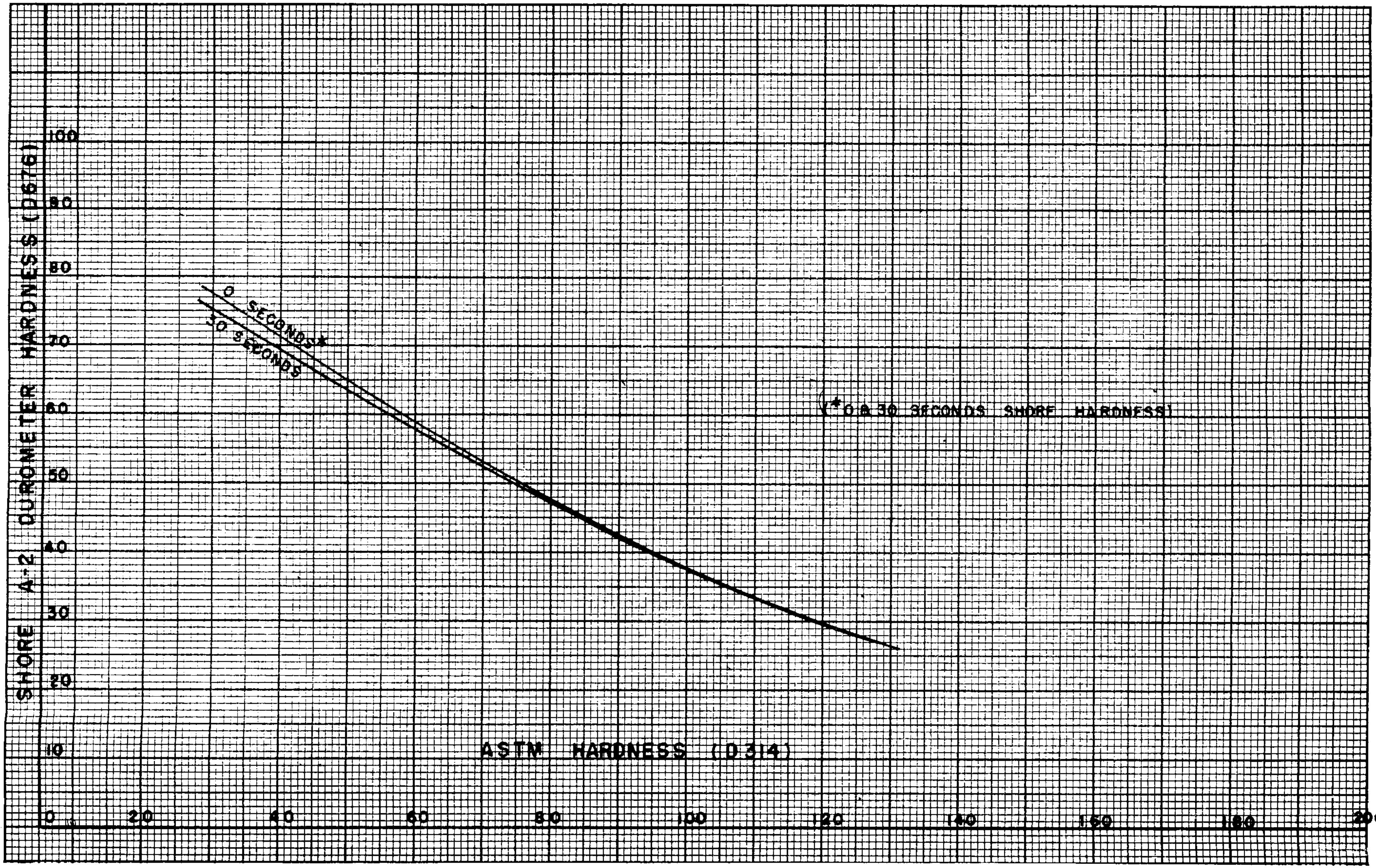


FIGURE 3 ASTM VS SHORE HARDNESS OF SILICONE RA-28 (METHYL VINYL POLYMER)

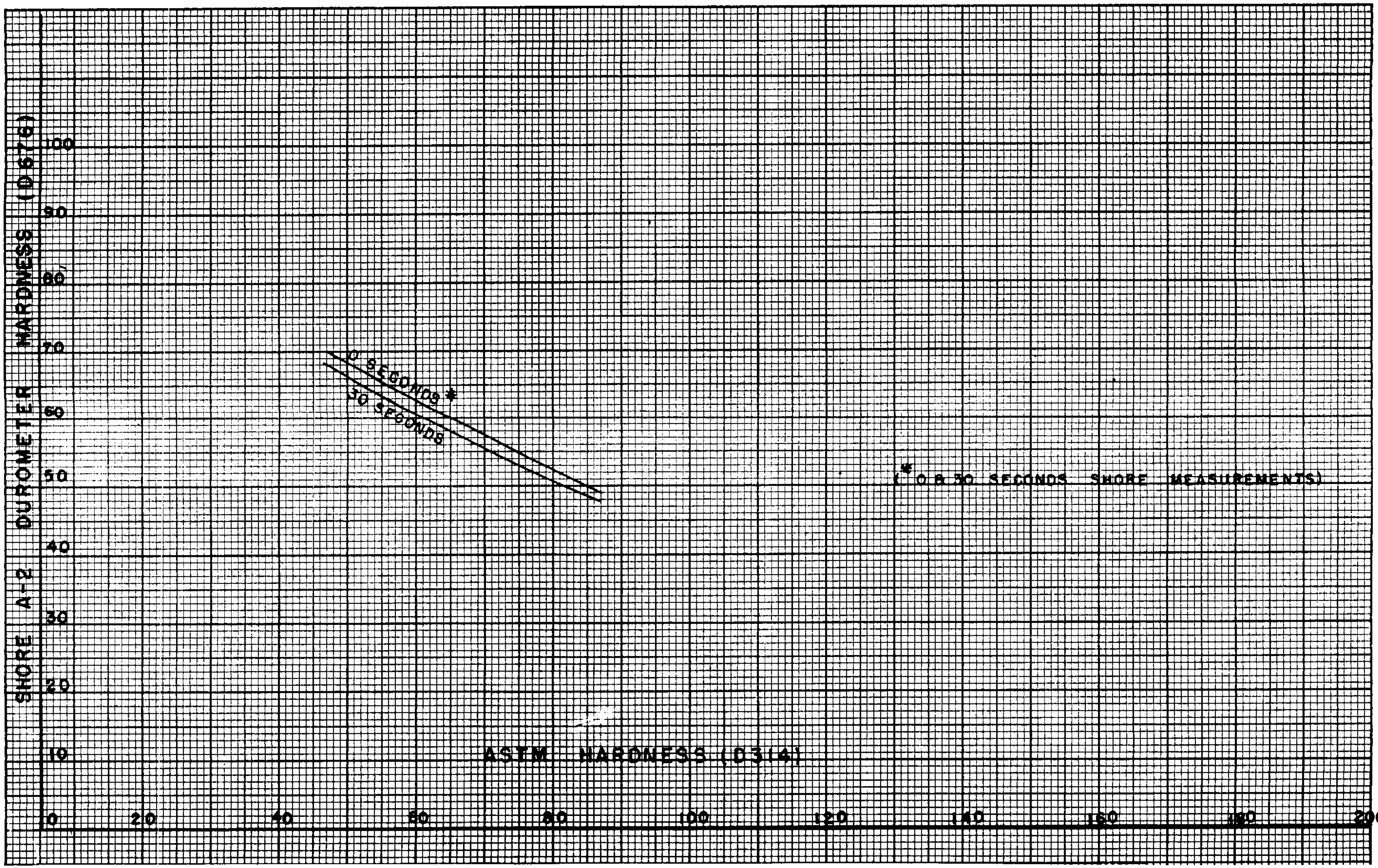


FIGURE 4 ASTM VS SHORE HARDNESS OF BUTYL (ISOBUTYLENE-ISOPRENE COPOLYMER)

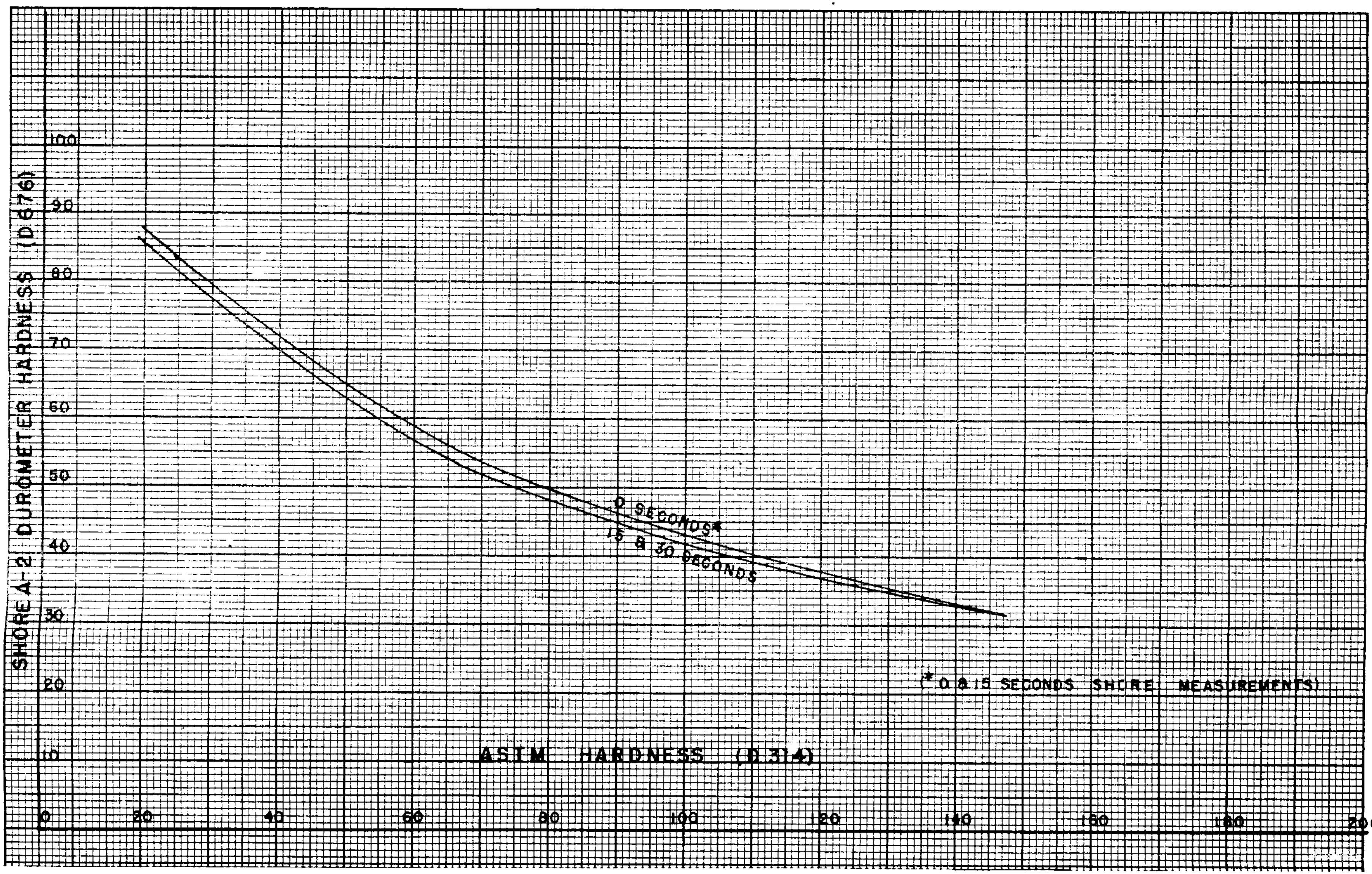


FIGURE 5 ASTM VS SHORE HARDNESS OF NEOPRENE (POLYCHLOROPRENE)

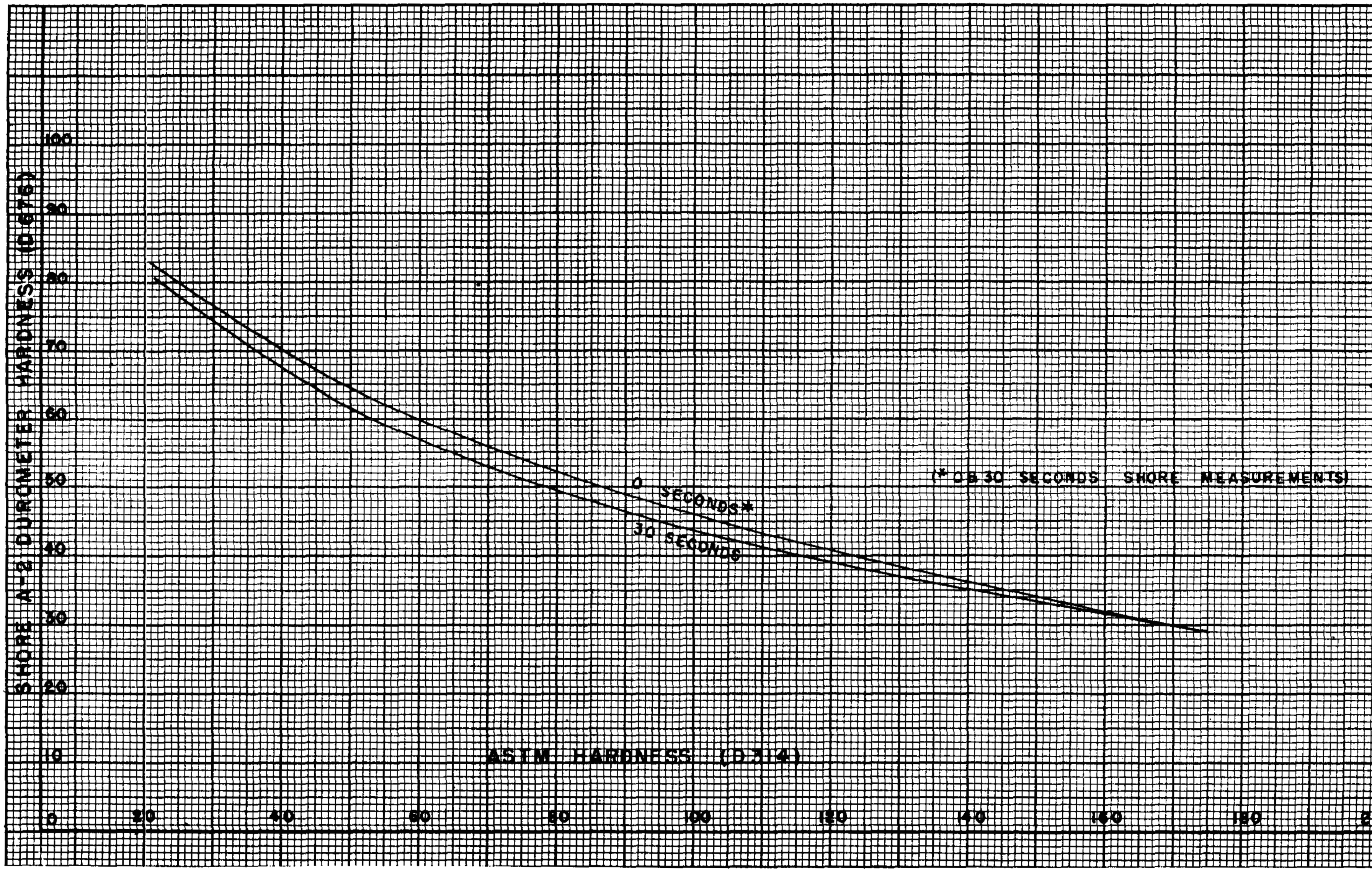


FIGURE 6 ASTM VS SHORE HARDNESS OF NATURAL (POLYISOPRENE)

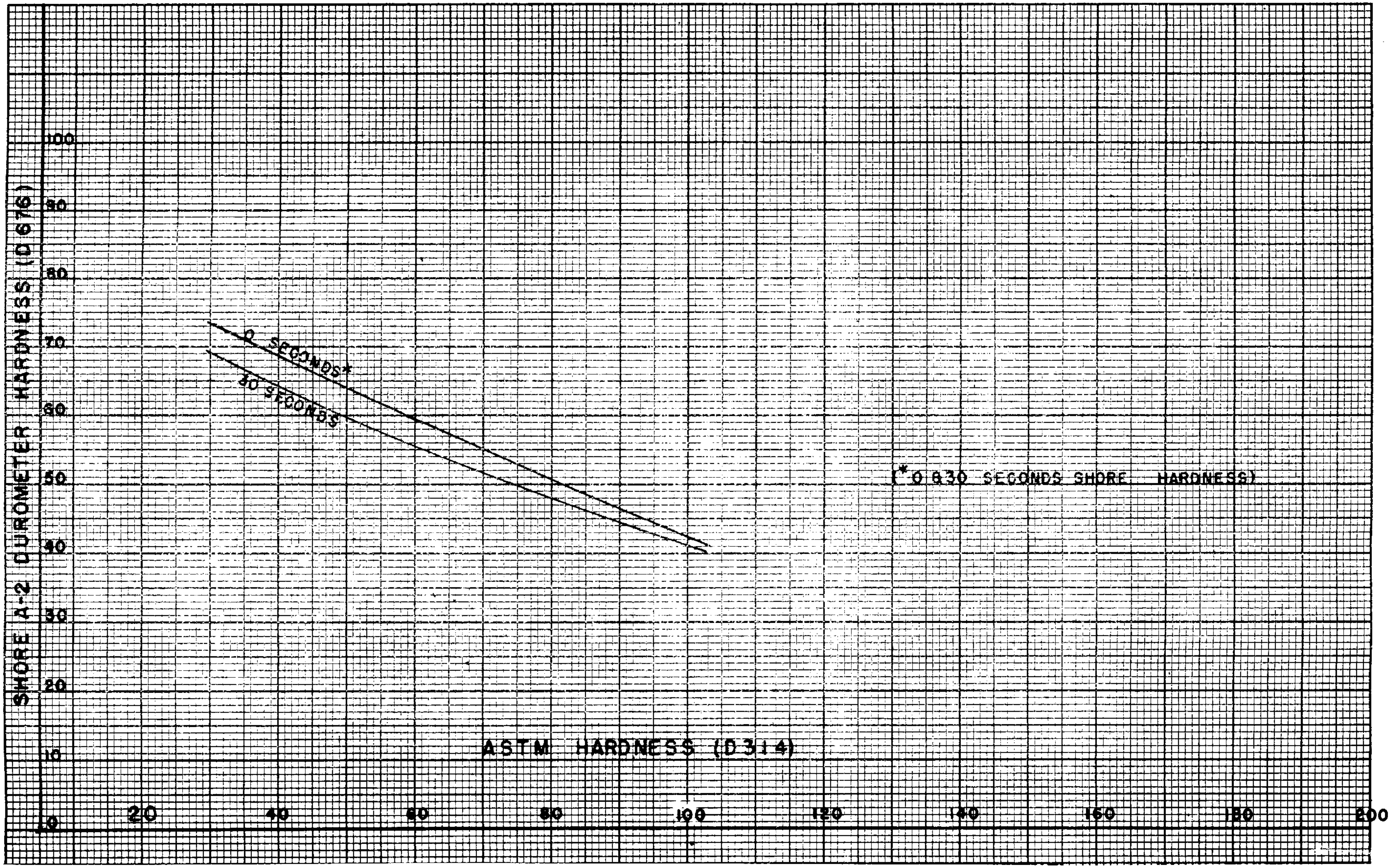


FIGURE 7 ASTM VS SHORE HARDNESS OF ACRYLIC (ACRYLIC ESTER COPOLYMER)

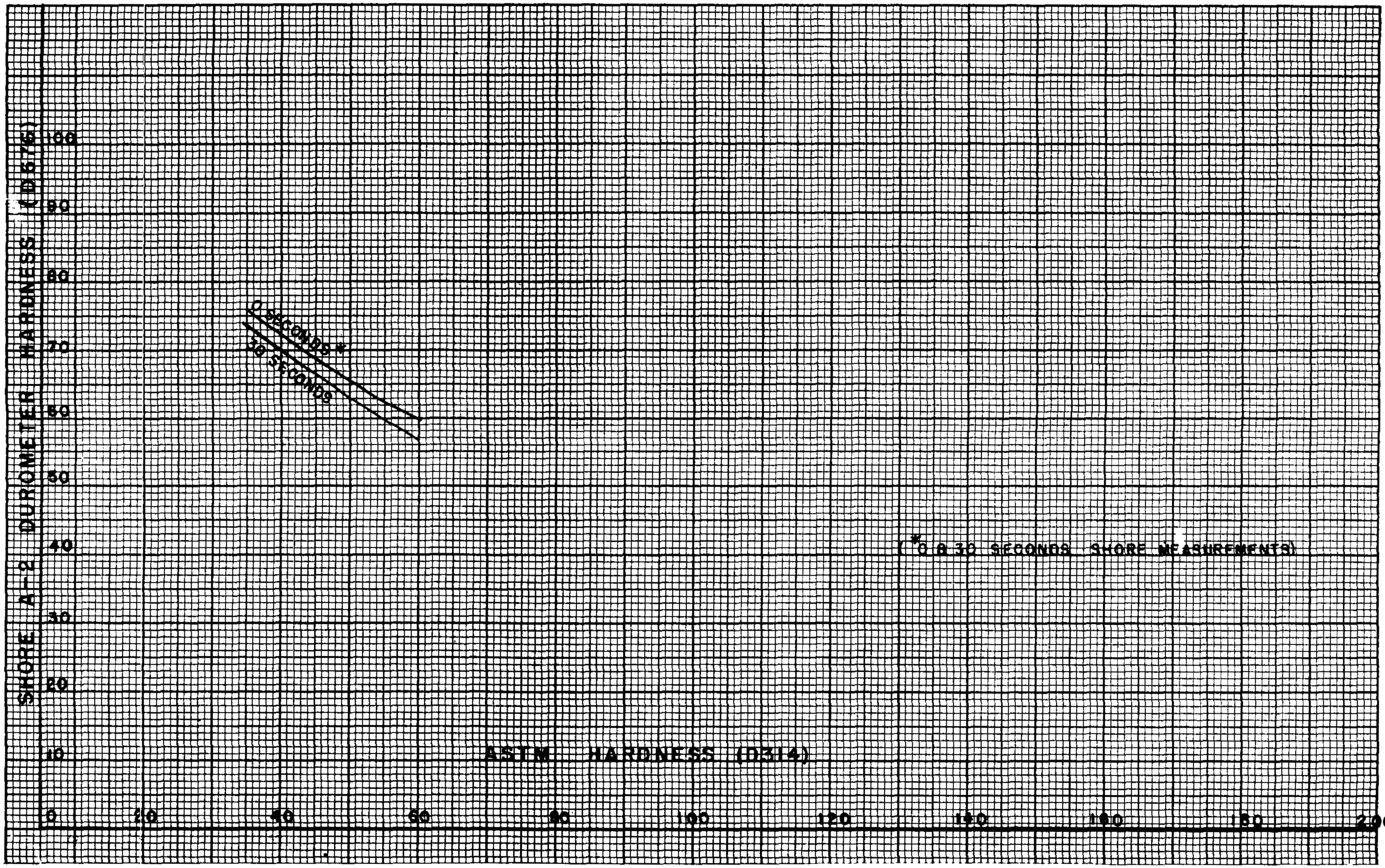


FIGURE 8 ASTM VS SHORE HARDNESS OF LS (METHYLTRIFLUOROPROPYL SILICONE)

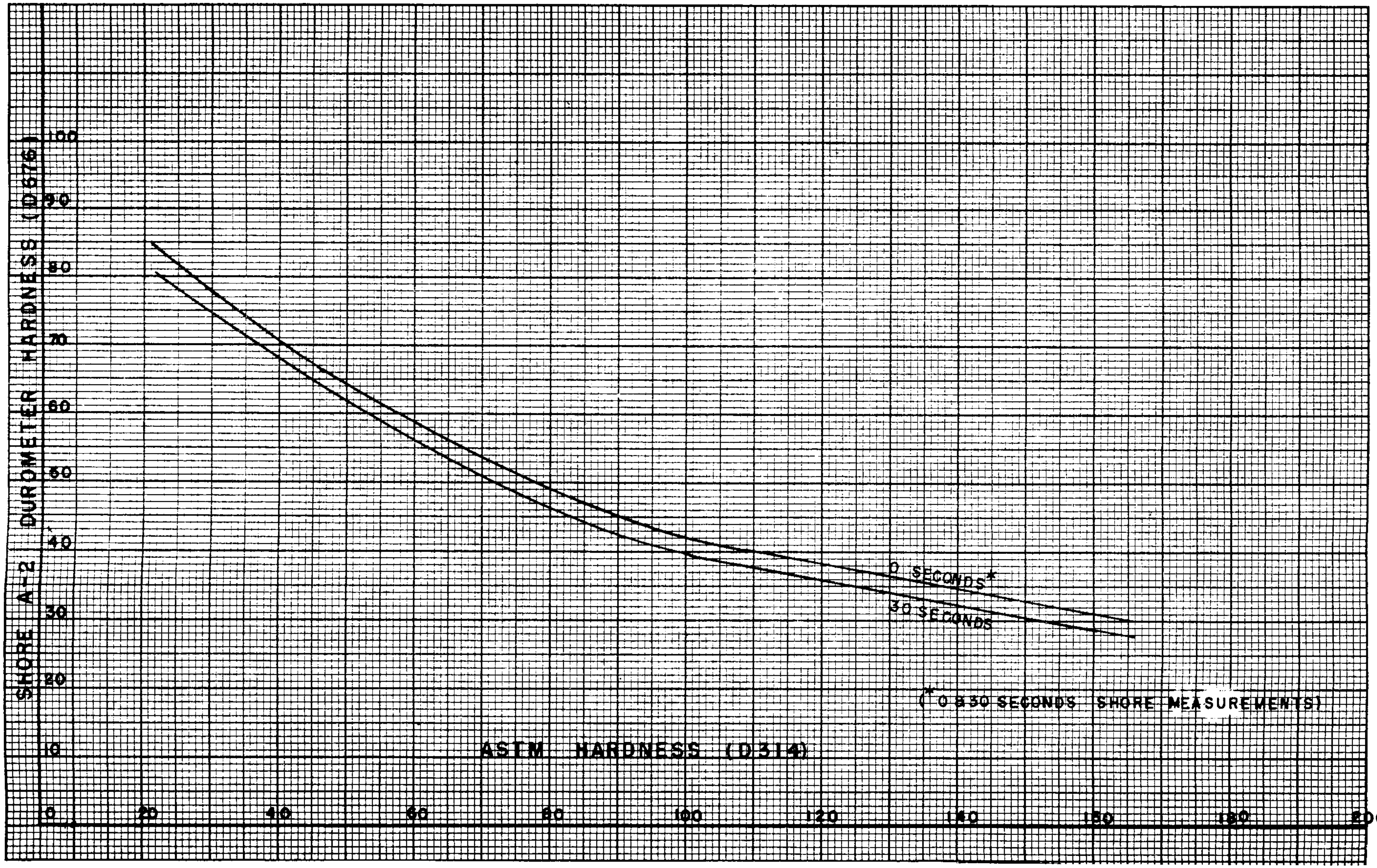


FIGURE 9 ASTM VS SHORE HARDNESS OF SBR (STYRENE BUTADIENE)

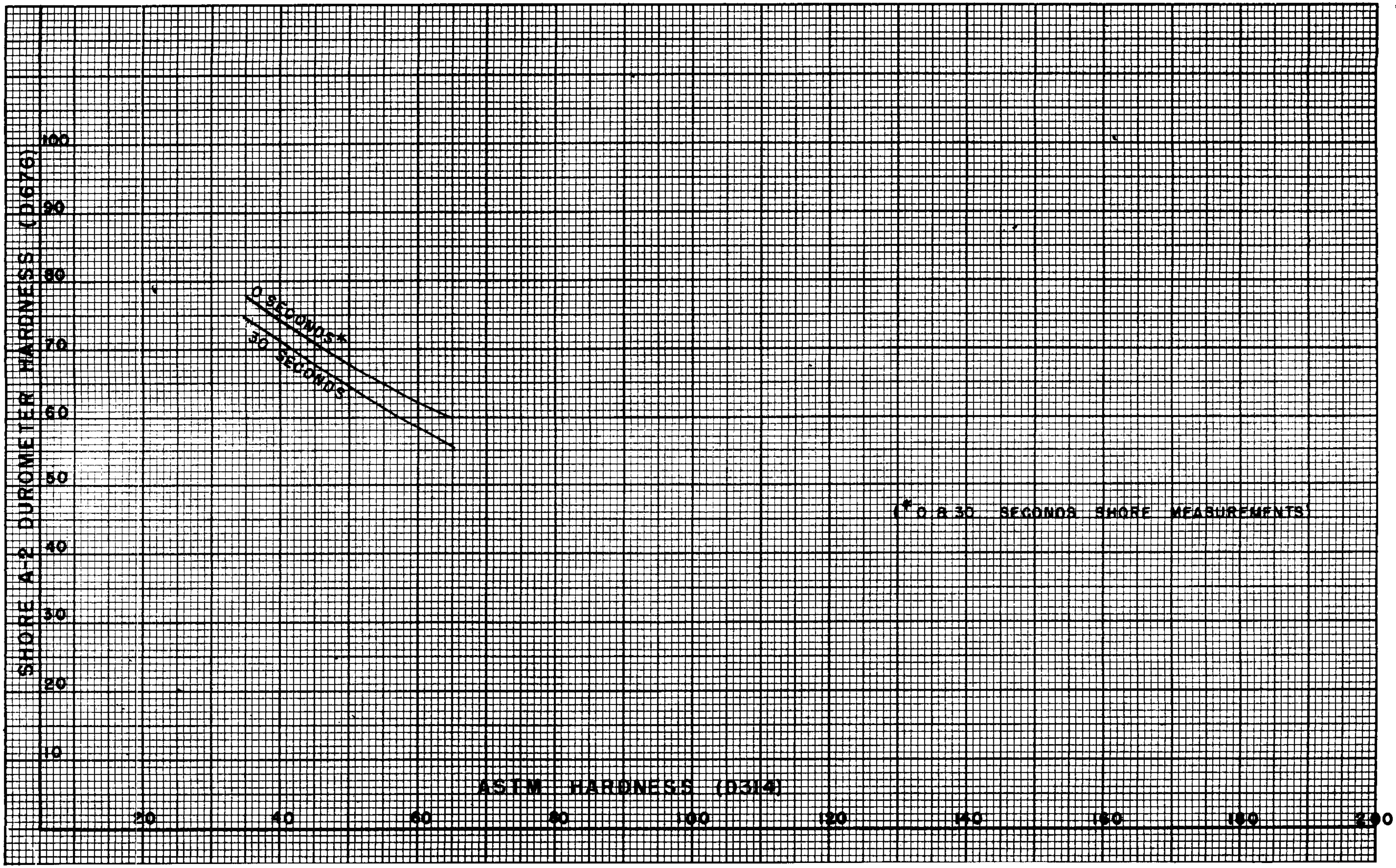


FIGURE 10 ASTM VS SHORE HARDNESS OF HYPALON (CHLOROSULFONATED POLYETHYLENE)

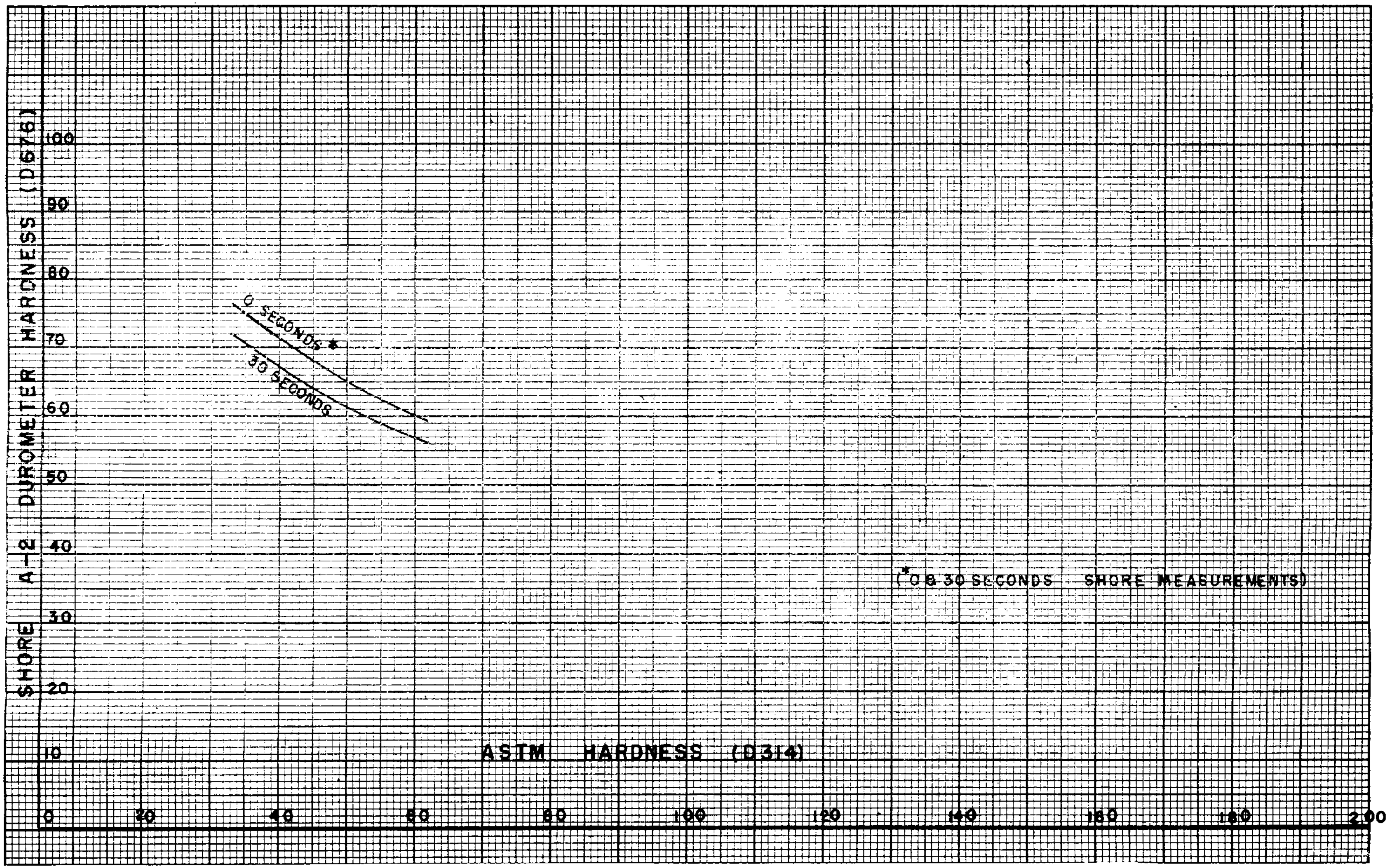


FIGURE 11 ASTM VS SHORE HARDNESS OF KEL-F (COPOLYMER OF VINYLIDENE FLUORIDE AND CHLOROTRIFLUOROETHYLENE)

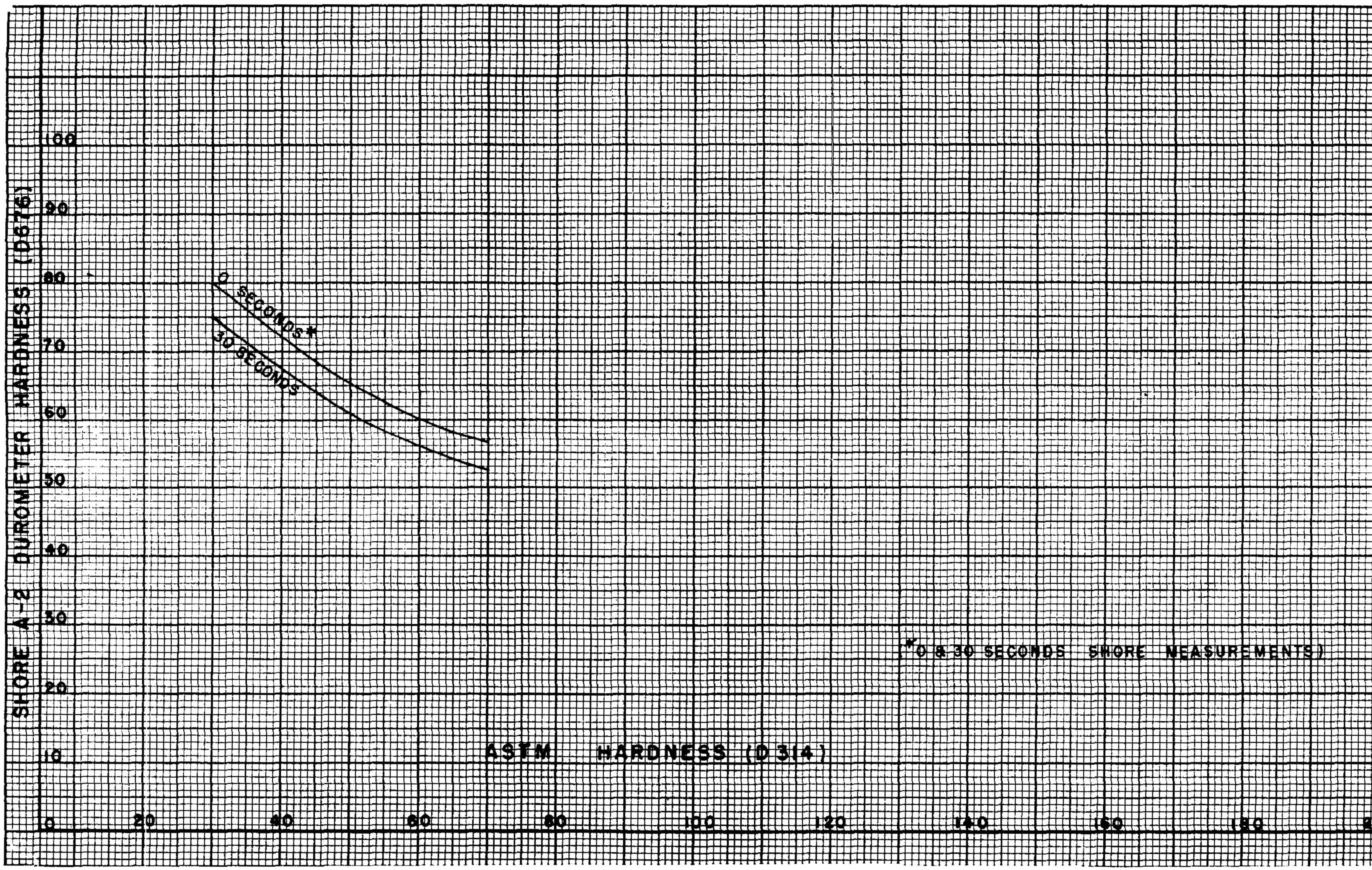


FIGURE 12 ASTM VS SHORE HARDNESS OF THIOKOL (POLYSULFIDE)

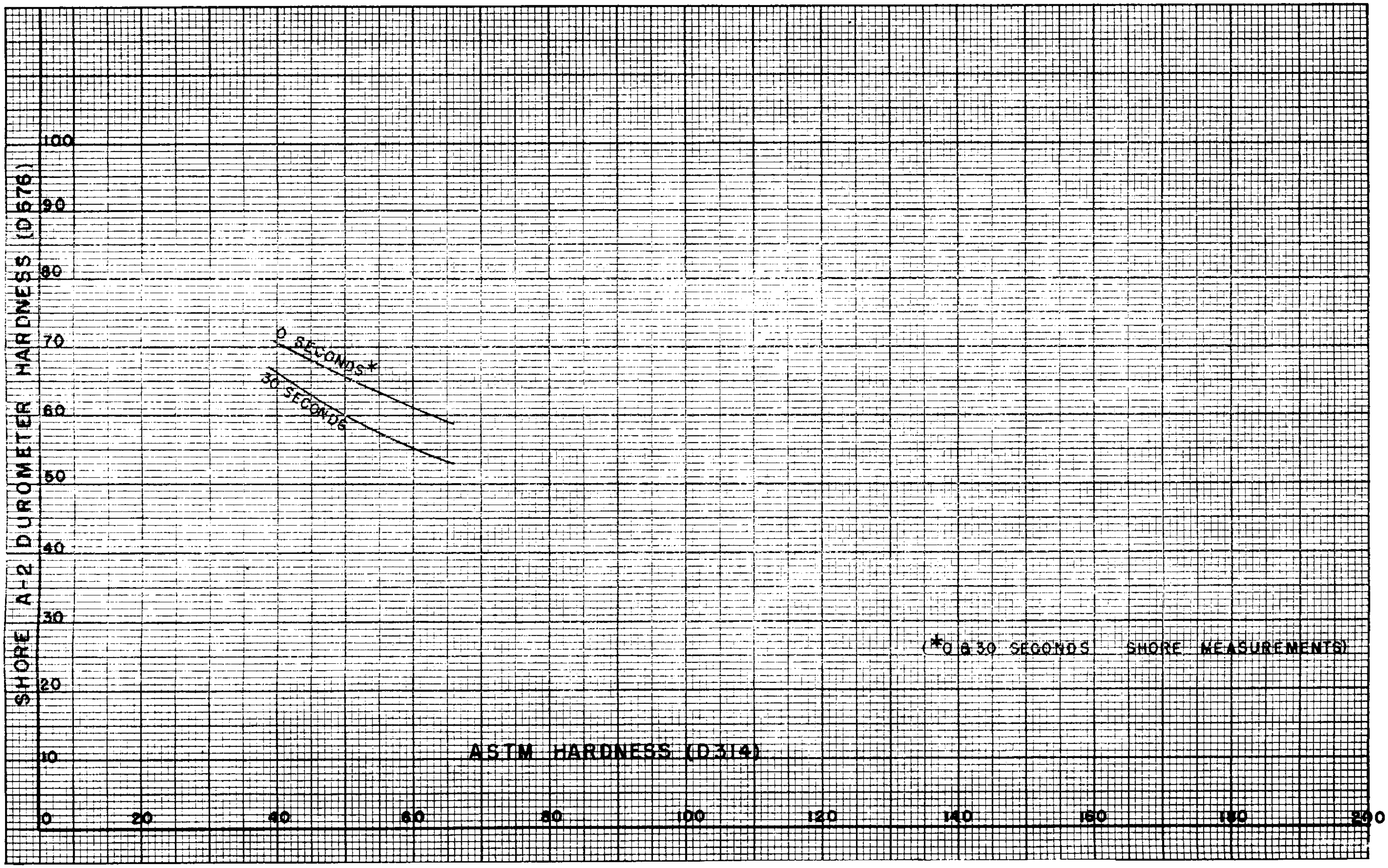


FIGURE 13 ASTM VS SHORE HARDNESS OF URETHANE (POLYURETHANE)

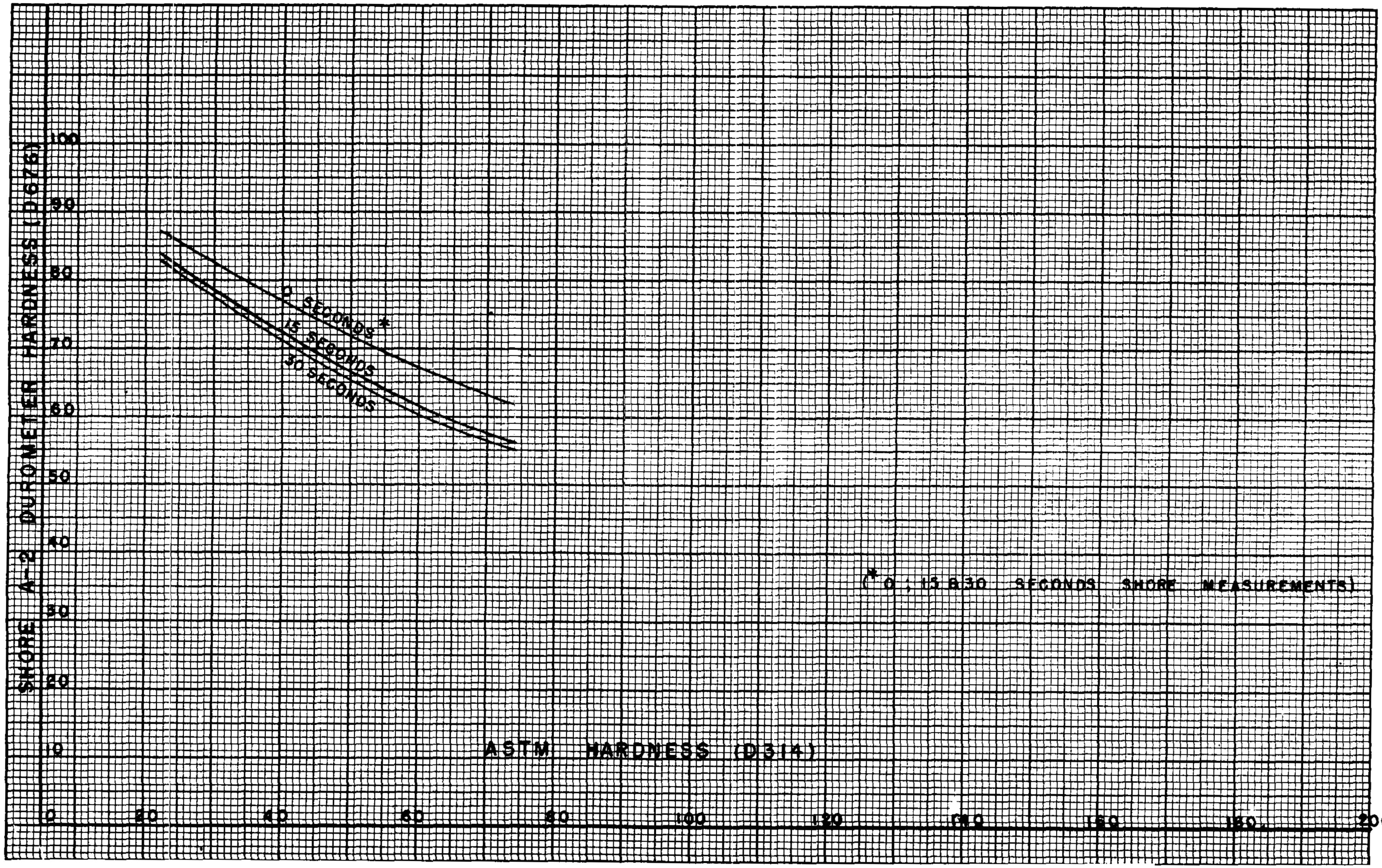


FIGURE 14 ASTM VS SHORE HARDNESS OF VITON-A (COPOLYMER OF VINYLIDENE FLUORIDE AND HEXAFLUOROPROPYLENE)

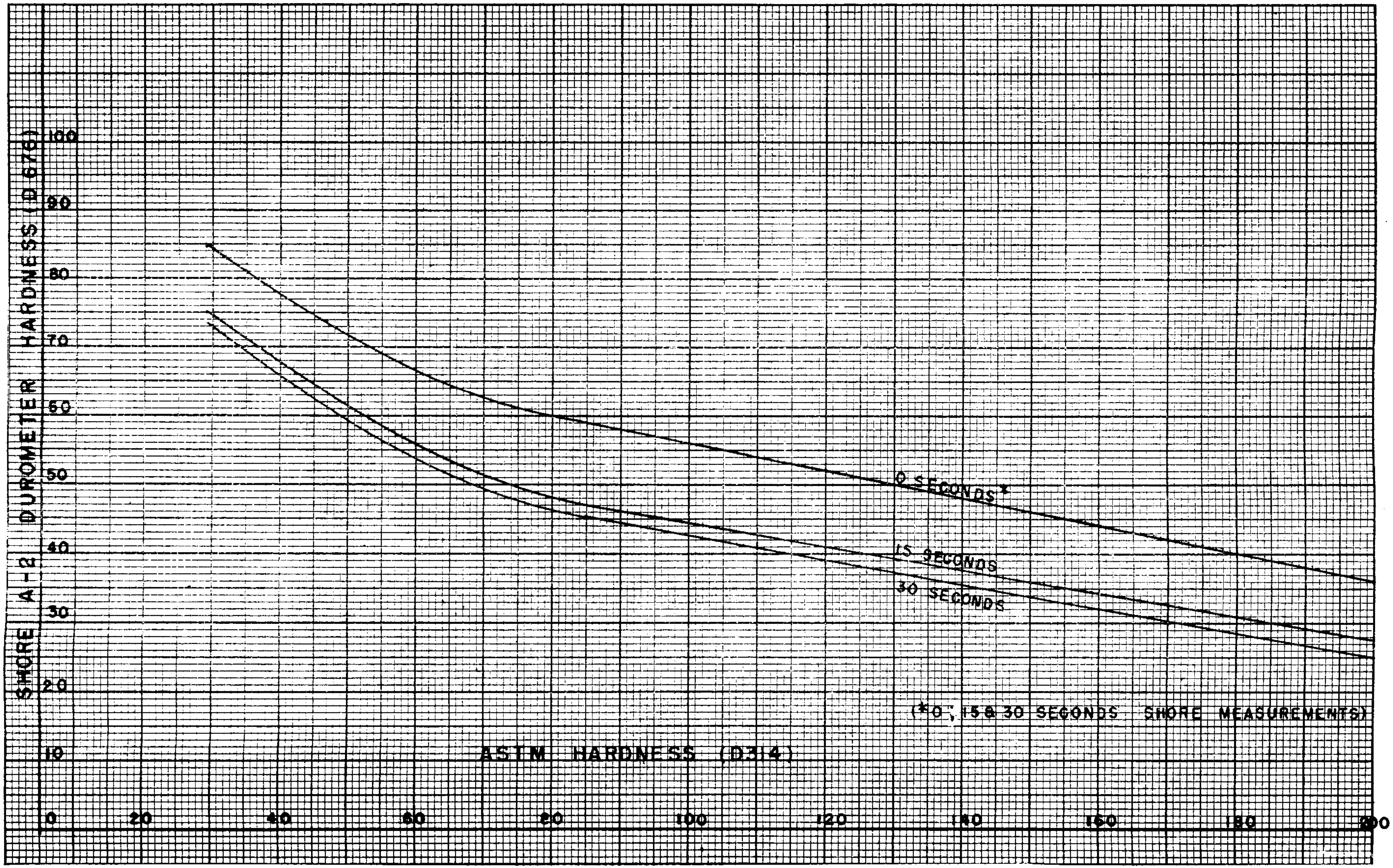


FIGURE 15 ASTM VS SHORE HARDNESS OF NBR (BUTADIENE-ACRYLONITRILE)

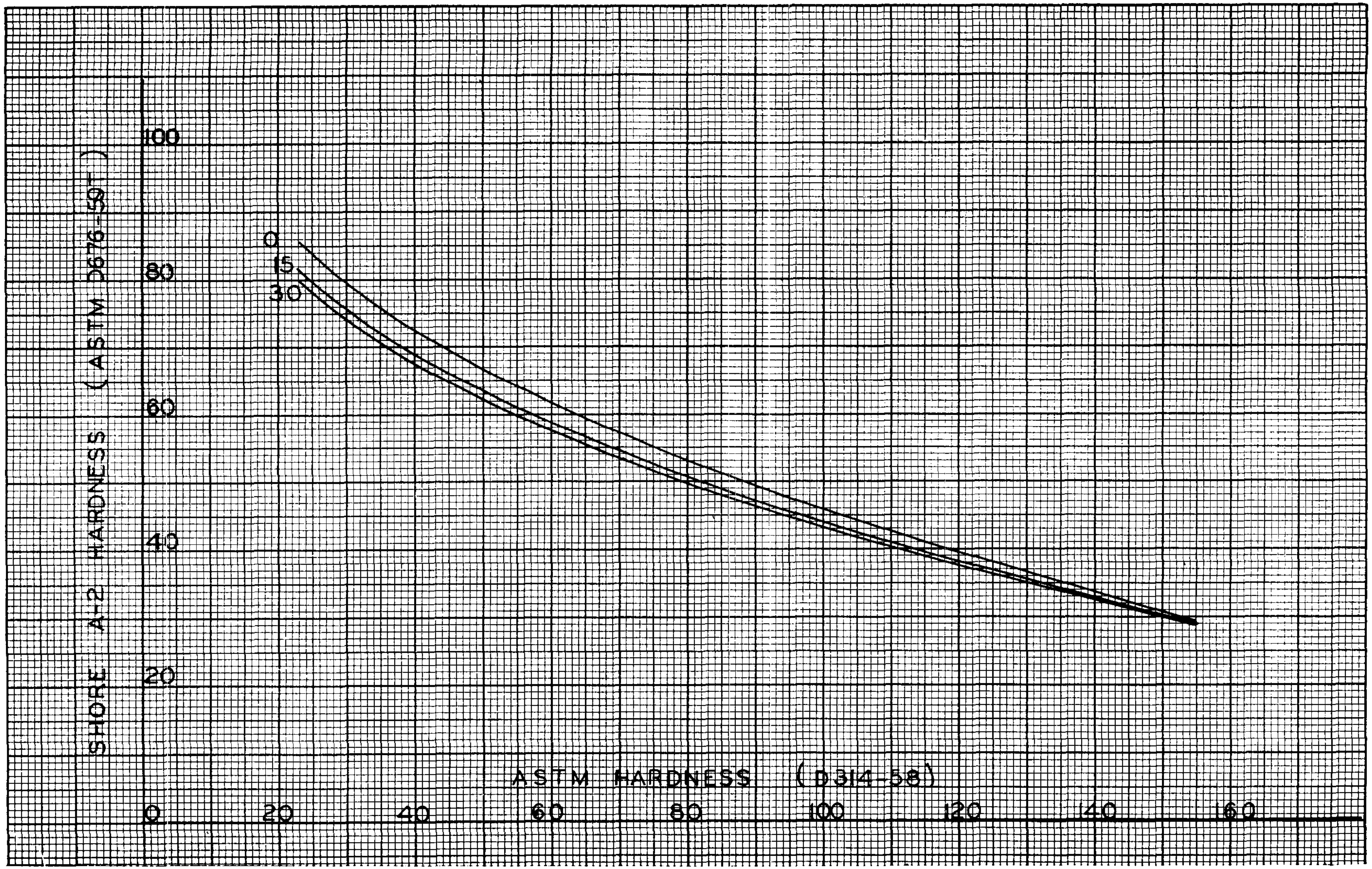


FIGURE 16 AVERAGE ASTM VS SHORE HARDNESS OF THIRTEEN COMPOUNDS AT VARIOUS TIME INTERVALS

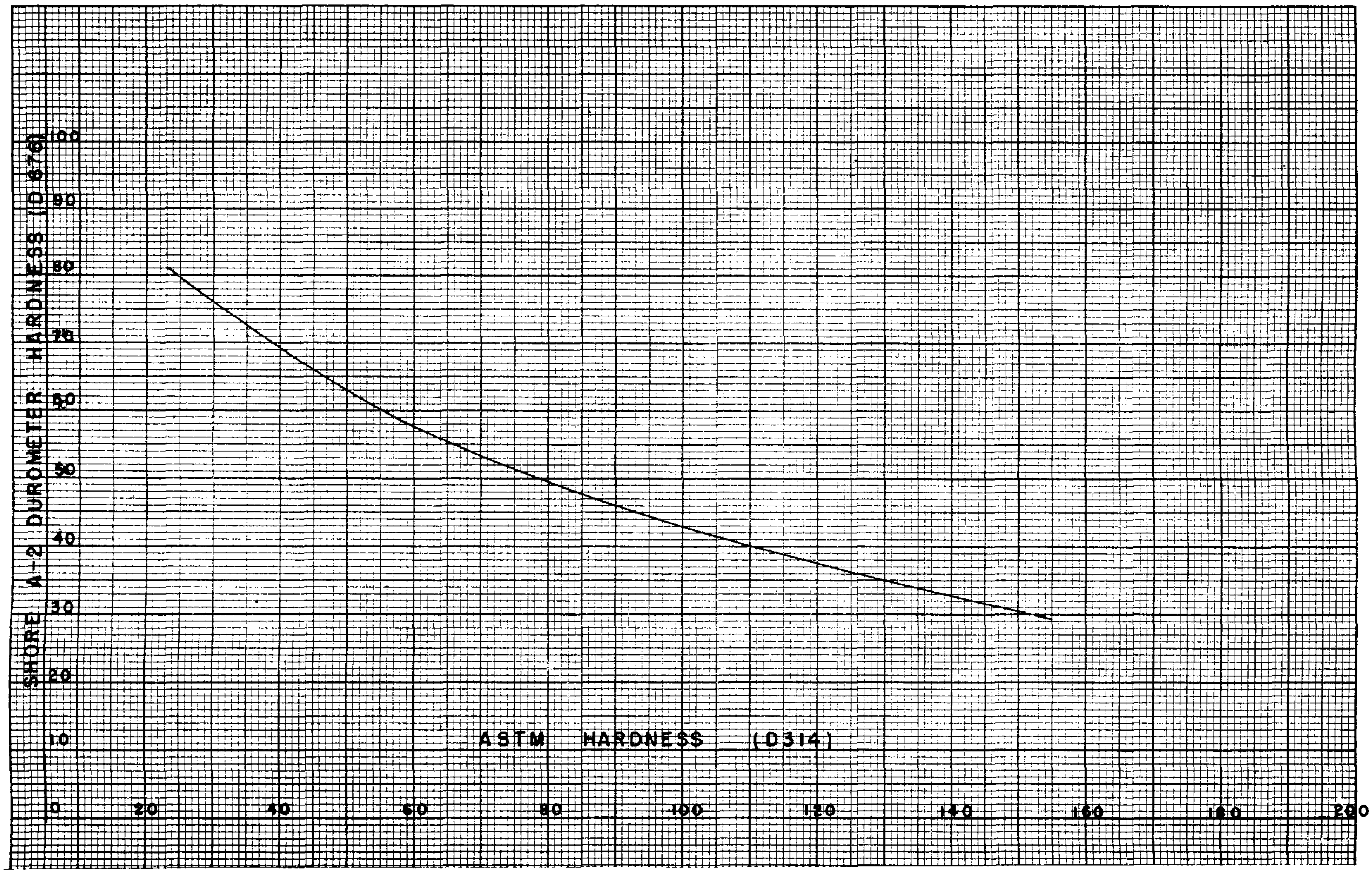


FIGURE 17 APPROXIMATE CORRELATION OF ASTM VS SHORE HARDNESS OF THIRTEEN STANDARD FORMULATIONS

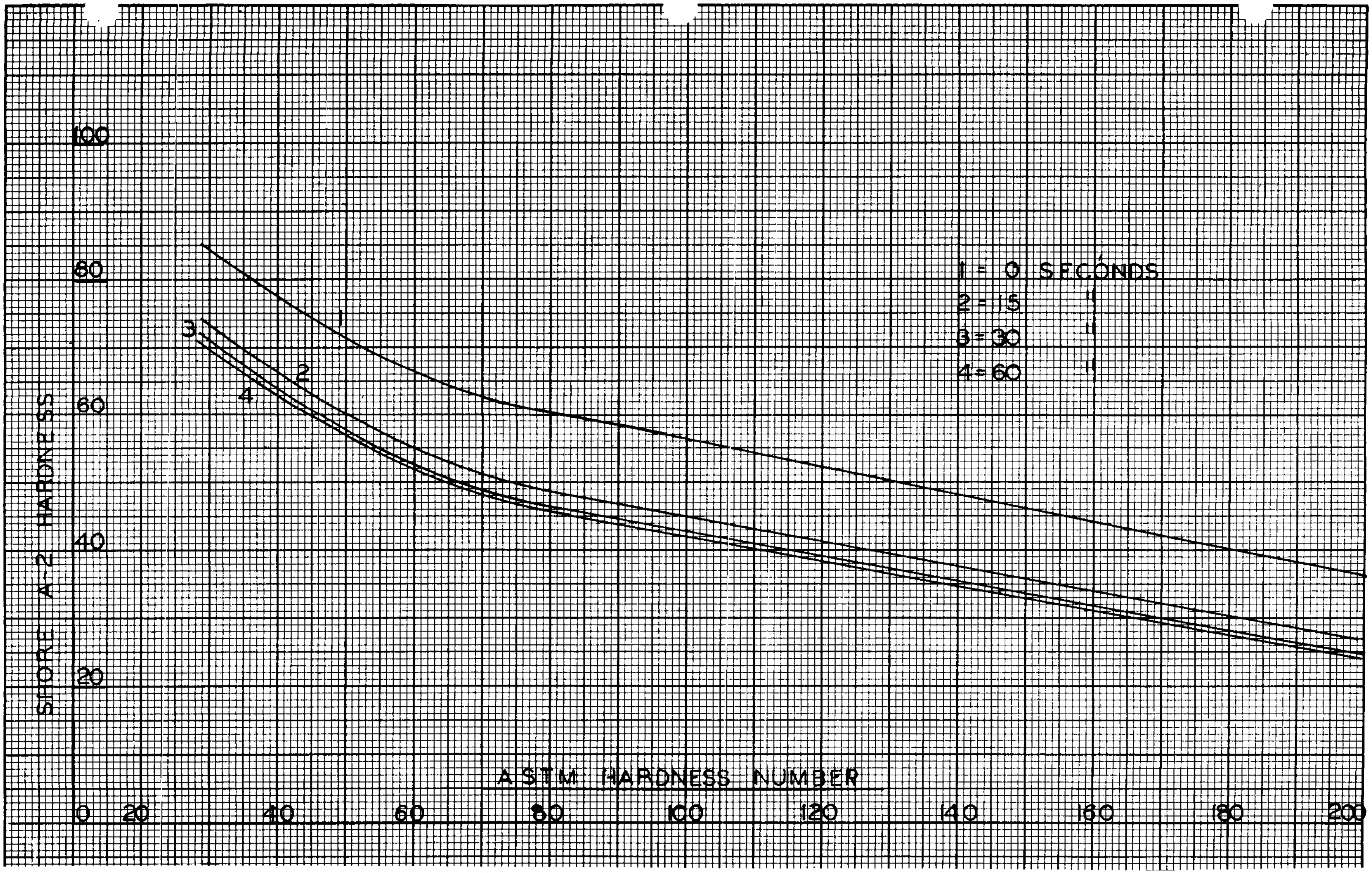


FIGURE 18 ASTM VS SHORE HARDNESS OF NBR COMPOUNDS AT VARIOUS TIME INTERVALS

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By

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The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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